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FOR
"HIGH PERFORMANCE MATERIALS IN COAL CONVERSION UTILIZATION"

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1.0 INTRODUCTION

This is the eleventh quarterly report on a three year grant regarding "High Performance Materials in Coal Conversion Utilization." The grant is for a joint university/industry effort under the US Department of Energy (DOE) University Coal Research Program. The University of Tennessee Space Institute (UTSI) is the prime contractor and The University of Pennsylvania and Lanxide Corporation are subcontractors.

UTSI and Lanxide have completed all the initially planned work envisioned at the start of the grant. In addition, Lanxide has produced and delivered to UTSI six innovatively manufactured DIMOX™ ceramic composite tubes. UTSI has added laser induced coating research to their originally planned work.

2.0 DISCUSSION

2.1 Objective and Scope of Work

The object of this grant is to test, analyze, and improve the heat and coal-slag corrosion resistance of a $\text{SiC(p)/Al}_2\text{O}_3$ ceramic composite tubular material. The material will be evaluated for its ability to withstand the pressures, temperatures and corrosion attack which would be encountered within a coal-fired high-temperature, high pressure air heater. The evaluation will include strength testing at elevated temperatures. The feasibility of several joining and coating techniques will also be investigated.

2.2 Task 1 - Materials (Lanxide Corporation)

Lanxide has completed all the work on their project regarding innovative forming methods for tubular preforms and six of these tubes were delivered to UTSI for testing and analysis. Two of the tubes were made from preforms which were fabricated by centrifugal casting using a mixture of 500 grit SiC particles and CERASET™ preceramic polymer. The remaining four tubes were produced with preforms fabricated by centrifugal low temperature molding (LTM) of an aqueous 500 grit SiC slurry. Towards the end of the program progress was made with the LTM approach. One obstacle not resolved is the controlled drying of the molded LTM preforms. The preforms might have been dried too quickly, which led to cracking of several preforms

2.3 Task 2 - Pre and Post Test Material Characterization

2.3.1 Subtask 2A - Strength of Materials Testing and Analysis (UTSI)

Three types of $\text{SiC}_{(p)}/\text{Al}_2\text{O}_3$ composite tubes recently manufactured using two new techniques by Lanxide were analyzed:

Tube A -- Commercially available (93-X-3063): tubular SiC preforms were fabricated by slip casting, e.g. the pouring of a SiC slip into plaster molds.

Tube B -- Being developed: tubular SiC preforms were fabricated by centrifugal casting using a mixture of 500 grit SiC particles and CERASET™ preceramic polymer.

Tube C -- Being developed: tubular SiC preforms were fabricated by centrifugal low temperature molding of an aqueous 500 grit SiC slurry.

The measured tube dimensions are listed in Table 1.

Table 1. Tube Dimensions

Tube	Outside Diameter (mm)	Wall Thickness (mm)
A	51 ± 0.5	5.0 ± 1.0
B	37.80 ± 0.2	2.55 ± 0.4
C	37.57 ± 0.5	3.85 ± 0.4

A phase image analysis was conducted with a system that consists of an optical microscope connected with a video camera and a computer with the software NIH (National Institute of Health) image 1.52. Phase (chemical and physical) distribution and corresponding quantitative analysis was performed using optical images and Image 1.52 software. Table 2 summarizes the analysis results. Figures 1-3 are the microstructures within the tubes.

Table 2. The Measurement for Phase Volume Percent (%)

Tube	SiC	Al ₂ O ₃	Pore	Al
A	61.4	34.7	1.4	2.5
B	62.5	32.1	2.7	2.7
C	59.2	32.2	5.4	3.2

The experiment procedures used in this study were exactly the same as used by Peter LaRue⁽¹⁾ to analyze Lanxide tube materials. The test was conducted at ambient temperature. (Tube A was treated with an oxide coating whereas tubes B

Figure 1. A cross section view of tube A.

Figure 2. A cross section view of tube B.

Figure 3. A cross section view of tube C.

and C were not. The exposure testing was not done, therefore, on tubes B and C as the surfaces were not the same as on the tube A tests). The test specimen numbers of C-Shaped rings were 40 for tube B, and 12 for tube C. Table 3 gives a summary of the strength data obtained from this testing.

Table 3. Summary of Strength Results

Tube	σ_o Characteristic Strength	m Weibul Parameter	E Elastic Modulus
A	410 MPa	10.91	276 GPa
B	352 MPa	8.81	274 GPa
C	385 MPa	7.47	246 GPa

The new processing methods (B&C) appeared to provide a marginally higher precision for the wall thickness dimension compared with the old processing. However, the new methods result in producing higher porosity and at the same time decrease the strength.

2.3.2 Subtask 2B - Corrosion Thermodynamic Analysis (U of Pa)

Work this quarter has focused on complementing UTSI's laser coating work, as well as initial modeling of the Foster Wheeler HIPPS system.

As discussed in prior quarterly reports, UTSI has coated the DIMOX™ composite materials with an Al_2O_3 ceramic layer. Initial bending tests showed a 14% reduction in bend strength after coating. However, the long term kinetic and thermodynamic modeling predicts a possible increase in strength after several hundred hours of exposure at high temperatures. The reasons for this are as follows. The laser coating process may have introduced a larger defect population at the surface of the DIMOX™ specimens. This may be due to microcracking, volatilization, etc. However, after long exposure times, the scale/substrate interface moves into the DIMOX™ sample. As a result, the load bearing "outer surface" at long times is the same inner surface as for the uncoated specimens. Additionally, the modeling predicts the reaction:

should become prevalent after several hundred hours. Mullite has a higher melting temperature (1920°C) than pure silica (1770°C) and should be more resistant to hot

corrosion. The UTSI Al_2O_3 coating should encourage the formation of mullite during long time oxidation.

Modeling of the Foster Wheeler HIPPS SiC system continues. Work has focused on the oxygen poor "Gas" side, with the following compositions:

CO_2	18.6%
H_2O	7.6
N_2	71.3
O_2	2.5

Initial work has suggested that the equilibrium oxygen partial pressure may be below the necessary level for protective SiO_2 scale formation.

2.4 Task 3 - Exposure Testing

2.4.1 Subtask 3A - Bench Scale Lab Tests (UTSI)

This subtask is complete. It was originally planned that the innovatively produced tubes (tubes B and C) would be tested after exposure to the same corrosive environment as experienced by the production tubes (tube A). The material strengths could then be compared. The new tubes, however, were not oxide coated by Lanxide and therefore it would not be fair to compare strengths after exposure and so were not exposed. Two of the tubes (tube B) have been sent to Dr. Kristin Breder at ORNL and she may perform corrosion testing/analysis if she feels it would be beneficial.

2.4.2 Subtask 3B - Field Exposure Tests (UTSI)

The subtask is complete.

2.5 Task4 - Project Management

Mr. William Boss presented a poster session as part of this grant at the University Coal Research Conference in Pittsburgh, PA on June 4, 1996.

Mr. Chenghe Xiao has completed a Draft of the first two chapters of his dissertation entitled, "Laser Induced-Reaction Coating of Ceramics on Ceramics." This report will focus on the work that Mr. Xiao has performed under this grant.

UTSI has requested a no-cost grant extension of this grant beyond the September 1996 end date. This extension is needed to allow the detailed analysis of the coating results. The extension will also provide more time to ORNL and ANL to complete the work they offered to perform regarding the DIMOX™ material.

3.0 SCHEDULE

The laboratory work is now completed and all that remains is analysis and final reporting.

The U of Pa continues work on the modelling of the HIPPS environment on silicon carbide to predict the suitability of a ceramic heat exchanger. This will be completed soon and will be reported on in the final report.

Lanxide has completed the delivery of all the tubes called for in their contract and in addition, six innovatively produced tubes.

4.0 CONCLUSION

All that remains on this grant is the analysis and reporting of the results of the corrosion and strength testing of laser coated DIMOX™ materials and the results of the corrosion modeling of the Foster Wheeler proposed SiC HIPPS heat exchanger.

5.0 REFERENCES

1. P. F. LaRue, "An Evaluation of the Strength of a $\text{SiC}_{(p)}/\text{Al}_2\text{O}_3$ Ceramic Matrix Composite Exposed to Coal Slag at Elevated Temperatures," MS Thesis, University of Tennessee, Knoxville, TN, (1995).